BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents *will not* correct images, please do not report the images to the Image Problem Mailbox.

1 462 168

PATENT SPECIFICATION (11) 1 462 168

(21) Application No. 29855/75 (22) Filed 16 July 1975
(31) Convention Application No. 489278 (32) Filed 17 July 1974 in
(33) United States of America (US)
(44) Complete Specification published 19 Jan. 1977
(51) INT CL² A23L 2/38
(52) Index at acceptance
C6E 6D

(72) Inventors CORNELIS HENDRIKUS JAPIKSE, GYANENDRA SINGH and TIMOTHY JAMES KENNEDY

(54) RAPIDLY DISSOLVABLE DRY BEVERAGE MIX

(71) We, THE PROCTER & GAMBLE COMPANY, a corporation organised and existing under the laws of the State of Ohio, United States of America, of 301 East Sixth Street, Cincinnati, Ohio 45202, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a food product, more particularly a dry beverage flavour mix. Such a mix may be used in preparing a beverage by adding the flavour mix to a liquid, for example water, and dissolving the dry mix therein. These aqueous beverages may either be consumed as such, or, more typically, may be carbonated to provide sparkling, effervescent soft drinks.

The market for carbonated beverages has increased very considerably, and today this market supports an industry involving many millions of pounds in value. Up to the present, this market has been served almost exclusively by beverages which are precarbonated to the proper level for immediate consumption and then packed in bottles or cans. While carbonation of beverages at the point of consumption, such as soda fountains, has been practiced for years, preparation of point-of-consumption individual servings of carbonated beverages in the home has not thus far been widely accepted.

One method of making carbonated beverages in the home involves the dilution of flavour syrups to the proper concentration and carbonating the resulting solution, such as would be done at a soda fountain. However, such an approach is burdensome for the volume of carbonated beverages consumed in the home, involving the procurement and correct dilution of the flavour syrups, and hence has not met with widespread popularity. Another method of making carbonated beverages in the home involves the use of dry mixes containing flavours and sugars along with a gas generation system. This type of drink has also not gained wide consumer acceptance because products marketed thus far have not afforded the consumer a quality alternative to pre-carbonated, bottled beverages.

One large disadvantage of current dry beverages mixes is the relatively slow dissolution rate of the dry mix when added to water. Conventional dry beverage mixes often take up to 5 or 10 minutes to completely dissolve, and even then, some mixes leave an unsightly residue of undissolved particles or a semi-dissolved sticky material at the bottom of the drinking vessel. Also, conventional mixes typically require mechanical agitation to achieve complete dissolution and uniform concentration throughout the beverage. As is evident from the lack of substantial market volume, compared to pre-carbonated, bottled beverages, this extra preparation time and effort for conventional dry beverage mixes is extremely disadvantageous to the consumer who turns to a carbonated beverage as a readily accessible and convenient item of cold, liquid refreshment.

The present invention assists in making possible the formation of a dry beverage mix having an extremely rapid dissolution rate upon contact with water and requiring only a minimum amount of agitation for dissolution in an aqueous solution to form a beverage in a short period of time.

The invention also assists in making it possible to furnish the consumer with a convenient method for preparing carbonated beverages equal in concentration and quality of flavour and carbonation to pre-carbonated, bottled beverages.

According to the invention a process for preparing a rapidly dissolvable dry beverage flavour mix comprises:

(a) forming a mixture of beverage flavour base and sugar such that the mixture has a weight ratio of flavour base:sugar of from 0.03:1 to 0.15:1 and a total solids content of from 75% to 95% by weight;

(b) drying the mixture to form a stable foam, and

(c) grinding the stable foam to form discrete dry beverage flavour particles.

As used herein, the term "flavour base" is intended to cover all flavouring materials present in a beverage with the exception of the sweetening agent and any acid. While both the sweetening agent and the acid do influence markedly the flavour of a given beverage, the characteristic flavor results from the flavoring materials added. These flavoring materials come in the form of alcoholic extracts or essences, aqueous solutions and emulsions, solutions of flavors in glycerol and propylene glycol, and fruit-juice concentrates. Beverage flavor ingredients of an essential-oil character often require extraction with alcohol from the source raw material. Water-soluble flavoring components can be made as aqueous solutions. When necesary, due to the low water solubility, emulsions can be prepared or solutions in glycerol or propylene glycol can be made. Such emulsions or solutions avoid the use of alcohol. Concentrated fruit juices are used in preference to fruit juices themselves, for on dilution with water, greater fidelity of fruit flavor can be obtained. Also, as used herein, the term "sugar" is intended to be generic to cover not only sucrose, the sugar most commonly used in the production of beverages, but also includes dextrose (D-glucose), fructose (levulose), liquid sugar (either sucrose or an invert-sucrose blend), invert sugar (a 50—50 blend of dextrose and fructose), lactose or maltose.

The flavoring materials are typically used as a flavor concentrate, or "flavor base", of a given concentration. A beverage is obtained by diluting the flavor base to the proper strength for a particular beverage and incorporating the proper sugar content for that beverage. Of course, if the beverage will conventionally be mixed together in a dry mix which is then dissolved in the proper amount of water. A carbonated beverage may be obtained by adding such a dry mix to precarbonated water, or by adding the dry mix to plain water, such as tap water, and then carbonating this aqueous solution.

Formulations for various flavor bases are well known to those skilled in the art and may readily be obtained from published sources. For example, Merory, *Food Flavorings*, The Avi Publishing Co., 1960) Westport, Connecticut, U.S.A. lists various flavor base formulations in Chapter 17, entitled "Syrup and Soda Flavorings"; also in Jacobs, M.B., *Manufacture and Analysis of Carbonated Beverages*, Chemical Publishing Company, New York, N.Y., U.S.A. (1959), formulations for many fruit and non-fruit flavor bases are given. Complete flavor bases are also commercially available in standardized concentrations for ease in dilution to the proper concentration for the final beverage. These are commonly known as "2-ounce" or "4-ounce" flavor bases depending upon the amount of the base which must be added to a gallon of simple syrup which is then diluted with water to the final beverage strength. (As those skilled in the art will appreciate, a simple syrup is a solution of sugar in water, usually in a standard concentration). Commercially obtained flavor bases are typically about 50% solvent, usually water or ethanol, with the flavoring material typical for a particular flavor dissolved therein.

In accordance with the present invention, the flavor base, containing all the flavoring materials necessary for a particular beverage, is admixed with the total sugar content required for that amount of flavor base to form a thoroughly mixed homogeneous slurry. It has been found that in order to obtain the rapidly dissolving dry mix particles of the present invention, it is necessary that the flavor base/sugar slurry have a total solids content of from 75% to 95%, by weight, and preferably, from 85% to 94%, so that the beverage mix produced from the slurry is able to be dried in the proper physical form to achieve rapid dissolution of the particles. In this regard, it is often necessary to add a small amount of water to achieve the best consistency for admixing. Only so much water as is necessary for these purposes should be added since it is important that the total solids concentration be maintained in the range as set forth above. If too much water is added to the mixture, the flavoring materials in the flavor base will separate into oil and water layers resulting in a non-uniform flavored product. At the other extreme, at moisture levels of below 5%, the necessary dispersion of flavor base and sugar does not take place.

In the practice of the present invention, it is also important that the flavor base: sugar ratio, by weights, be from 0.03:1 to 0.15:1, in order to assure the production of rapidly dissolving flavor particles. While the specific optimum ratio will vary for different flavoring systems due to the different bases and flavoring materials used, the ratio for all known flavoring systems will fall within the above range. Thus, any specific optimum ratio can easily be determined by one skilled in the art in the light of the present specification.

The flavor base/sugar mixture is blended together to provide a slurry, or "slush", before drying. It has been found that any type of conventional mixing means may be used for blending the mixture as long as a thorough mixing is achieved. It is preferred, however, that the mixing means employed does not beat into the slurry an abundance of air since such an abundance of air will cause an unstable foam during drying and correspond-

ingly, a very low density in the final dried product. Also, it is preferred that the temperature of the slurry be kept below about 100°F during admixture to avoid any loss of volatiles or heat degradation of flavor materials. With these considerations in mind, it is thus recommended that low-shear mixing means be employed, since these will not beat in an abundance of air and will impart less energy to the mixture, and correspondingly, less heat.

If a commercially available beverage flavor base is used for the preparation of the dry beverage mix of the present invention, various substances may optionally be added to the mixture in processing it in accordance with the present invention to provide such a dry beverage mix. The only criterion that limits the possible inclusion of any optional ingredient is that it must be acceptable for use in edible food products. Other than this restriction, only the appearance of an undesirable off-taste or off-color for a particular beverage will place a practical limitation on the addition of any desired material. As those skilled in the art will appreciate, the variety of different materials which may be added is extremely broad indeed. For example, the present invention is concerned with a dry beverage mix which will be dissolved in water at the point of consumption. In doing this, the consumer will most likely use tap water which is slightly alkaline due to the mineral salts dissolved therein. Thus, to neutralize this alkalinity, an edible acid may be needed to achieve the same beverage pH as that achieved when distilled or de-ionized water is used as the beverage base. Any common food acid, such as citric, malic, or phosphoric may be used for this purpose. Of course, if phosphoric acid is used, it must be properly dried, as will be detailed hereinafter. Other materials which may be used include various additional flavoring ingredients which may be added to the commercial flavor base to modify its flavor, accentuate any especially preferred flavor notes, or replace any flavor notes which may be volatilized and lost during processing into a dry mix. Some of the more common flavoring ingredients which can be added to common beverages, such as a cola beverage, include, for example, extract of coca leaves, neroli oil, lime oil, lemon oil, orange oil, nutmeg oil, vanilla extract or cassia oil. Other flavoring materials for colas or any other desired beverages may be found in published formulation recipes for the particular beverage flavor desired. Other types of materials, such as food colorings, for example, certain artificial food colors or caramel coloring: stimultants, for example, caffeine; artificial sweeteners, for example, saccharin; bodying agents, for example, sorbitol or sodium carboxymethylcellulose; foaming agents, for example, licorice root extract or saponin-bearing extract of soaproot; or preservatives, for example, sodium benzoate, propylene glycol or ascorbic acid may, where permitted be added to the flavor base to achieve the respective desired results.

Alternatively, if the flavor base is formulated specifically for processing into a dry beverage mix in accordance with the present invention, standard flavor base formulations may be directly adapted for use by the addition of any desired optional ingredient not set forth in the standard formulation.

The mixture of flavor base, sugar and any optional ingredients is then dried to substantial dryness using conventional vacuum or freeze drying techniques to produce a dry beverage mix. As used herein, "substantial dryness" is the state at which the dried material feels dry to the touch, is free-flowing after being ground, and can be packaged similarly to conventional powders or granules without coating on or adhering to the package side walls. This will typically involve drying to a final moisture content of less than 3%, preferably, to less than 1%, and most preferably, to 0.1%, by weight. In keeping with the present invention, it is important that the slurry be dried by vacuum drying, or equivalent techniques, which cause the drying material to form a stable foam so as to form a beverage mix which is rapidly dissolvable in water requiring only a minimum amount of agitation. In the practice of the present invention, the slurry is placed in a relatively thin layer on a belt conveyor or on trays and dried under vacuum conditions. The slurry may be frozen before drying, but in any event, it is important that the integrity of the phyisical structure be retained during the vaporization of moisture. Specific vacuum drying techniques are known to those skilled in the art and will not be repeated herein for purposes of brevity. It has been found that best results are obtained if the slurry is dried within a period of 8 hours, and preferably within 3 hours, and that the temperature of the slurry is not raised above 100°F and preferably not above 50°F during the drying process. In the practice of the present invention, it has been found that conventional freeze drying techniques are equivalent to the vacuum drying techniques in the results obtained and thus may be advantageously employed. Freeze drying basically involves freezing to temperatures of about −10°F to about −40°F and removing the water by sublimation as the frozen slurry is gradually warmed under vacuum conditions. Again, any of the conventional freeze drying techniques known to those skilled in the art are suitable for the practice of the present invention as long as a stable foam is produced from the slurry during dehydration.

The dried product resulting from the dehydration step is then ground by conventional

size reduction techniques so as to make it more readily dissolvable when added to water and to enhance the appearance of the final product. Depending upon the fineness to which the particles are ground, they may either be screened to obtain groups of approximately uniform particle size, or the dried mix may be ground to a fine particle size and agglomerated using conventional agglomeration techniques. After grinding to a suitable particle size, the dry stable beverage mix particles are packaged in suitable aliquot portions for subsequent dissolution in a predetermined amount of water to obtain flavorful beverage ready for either carbonation or direct consumption.

The dry particles resulting from the practice of the present invention are unique in that a flavorful, sweetened beverage results within one minute after their addition to water and a minimum of agitation, whereas with conventional dry beverage mixes, prolonged mixing and a waiting time of at least several minutes is necessary before the mix is fully dissolved. The dy beverage particles of the present invention are further distinguished from conventional mixes in their appearance. Since the sugar crystals are uniformly coated with a flavor base/sugar mixture before drying, the dried product has a uniform color. Of course, the color will vary for different flavors and the food coloring added for the respective flavors, but due to the manner of drying the slurry, each flavor will retain a deep, rich color typical of that beverage flavor. This is in marked contrast to conventional dry mixes, which either have a "speckled" appearance due to the separate particles of sugar and colored flavor, or have a uniform "bland" appearance with no noticeable color provided for aesthetic appeal to the consumer.

Although we do not wish to be bound by any specific theory in the practice of our invention, it is theorized that the extremely rapid dissolution rate of the dry flavor particles of the instant invention is due to the unique slurry which is dried by conventional techniques. Since the flavor base is admixed with the entire beverage sugar content in a slurry having an unusually high solids content, it is theorized that only a portion of the sugar crystals dissolve and that this dissolved sugar/flavor base admixture uniformly coats the remaining sugar crystals. Upon drying, the sugar crystals are thus uniformly coated with a flavor base/sugar admixture in an amorphous physical form. This amorphous physical form is less structurally stable than the normal crystalline form of sugar and rapidly breaks down in the presence of water, or is thus rapidly dissolvable in water. This amorphous coating, or outer layer, would also account for the uniform texture and rich, darker appearance of the dry particles of the present invention.

The dry particles of the present invention may be added to water and immediately consumed as a flavorful, sweetened drink, or may be made a part of a dry carbonated beverage mix. If the dry flavor particles of the present invention are to be used in a dry mix adapted for making carbonated beverages, the particles may be advantageously employed with a variety of carbonation systems to provide suitably carbonated, flavorful beverages. For instance, the dry beverage particles are disclosed herein can be added to pre-carbonated water or, the dry beverage particles can be combined with an economical point-of-consumption carbonation system and this combination then added to tap water to provide a convenient, carbonated beverage. Examples of suitable sources of pre-carbonated water are the use of bottled precarbonated water, commonly known as "club soda" or the use of pressure carbonators which utilize CO_2-charged cylinders to carbonate water as it is dispensed, such as is done at a soda fountain. Examples of point-of-consumption systems which utilize regular tap water for the beverage include the use of a "chemical couple" such as those disclosed in U.S. Patent Specification No. 3,241,977, or U.S. Patent Specification No. 3,492,671; or the use of CO_2-loaded zeolite molecular sieves, such as that disclosed in British Patent Specification No. 1,382,896.

The latter-named carbonation system i.e. the use of CO_2-loaded zeolite molecular sieves, is especially preferred in the practice of the present invention. Molecular sieves of this type are crystalline aluminosilicate materials of the following general formula:

$$M_{2/n}O.SiO_2.aAl_2O_3.bH_2O$$

in the salt form, where n is the valence of a metal cation M. M ordinarily is Na or K but may be other cations substituted, by exchange, a is the number of moles of alumina, and b is the number of moles of water of hydration. Due to the crystalline nature of such materials, the diameters of the surface cavities and of the internal pores are substantially constant and are of molecular magnitude. For this reason, the crystalline aluminosilicates have found wide use in the separation of materials according to molecular size or configuration, hence the name molecular sieves. Upon removal of at least some of the water of hydration by heating, the crystalline aluminosilicates become highly porous and are characterized by a series of surface cavities and internal pores which form an interconnecting network of passageways within the crystal. Such dehydrated molecular sieves are often referred to as "activated", meaning that they are ready to selectively

absorb molecules of a given size. Carbon dioxide is strongly absorbed, or "loaded", on such sieves, but can subsequently be displaced by the stronger and preferential absorption of water. Hence, the release of absorbed CO_2 from molecular sieves upon contact with an aqueous potable liquid provides a convenient and economical point-of-consumption beverage carbonation system. By employing molecular sieves loaded with carbon dioxide to the extent of at least 5% by weight of the molecular sieves and by carbonating a beverage at temperatures of from about 35°F to about 70°F (the temperature of the beverage) and atmospheric pressures, suitably carbonated beverages can be obtained in about 1—10 minutes. As might be expected, increasing the quantity of molecular sieves employed for a given amount of beverage solution increases the amount of carbon dioxide released. Although the optimum amount of molecular sieves employed will vary with the characteristics of the particular sieve type employed and the beverage solution to be carbonated, the preferred quantity of molecular sieves ranges from about 0.5 gram to about 4 grams of molecular sieves (before loading) per fluid ounce of beverage to be carbonated.

It is important that the dry flavor particles of the present invention be packaged in a moisture-proof container, in that the particle physical structure collapses when wetted, due to the extremely rapid dissolution rate. A convenient method for ensuring that these flavor particles are not degraded by exposure to moisture during storage periods is to have them packaged in the presence of a desiccant. The CO_2-loaded molecular sieve disclosed in the above-mentioned British Patent Specification No. 1382896 is a suitable desiccant for such purposes. Thus, the presence of such CO_2-loaded zeolite molecular sieves in combination with the dry flavor mix has multiple advantages in that they provide protection of the flavor particles during storage, provide a convenient and sufficient carbonation system during use, and the effervescent action resulting from the molecular sieves immersed in water is sufficient to provide adequate agitation for the dissolution of the dry flavor particles of the instant invention.

If dry cola flavor particles are desired, it is desirable that a mono-alkali orthophosphate be added to the cola base containing phosphoric acid, in accordance with the teachings in British Patent Application No. 29856/75 (Serial No. 1462169).

The following Examples serve to illustrate various embodiments of the invention but are not intended to limit the invention in any way.

EXAMPLE I

To 242 parts of granulated sugar (baker's sucrose), 20 parts of a commercially available orange flavor base and 10 parts water were added and thoroughly mixed. The resultant slurry had a flavor base: sugar ratio of 0.083:1 and an 89% total solids content. The slurry was spread in a layer about 1/4" thick and placed in a Stokes freeze dryer at about 75°F. Without precooling, the slurry was dried for 3 hours at 75°F at 1 mm Hg absolute. The dry stable foam resulting from this operation was then ground and passed through U.S. Standard Sieve screens of 7 and 12 mesh to select particles of the desired size range.

Twenty-five grams of the dry flavor particles were then placed in an 8-ounce glass, along with an operative amount of CO_2-loaded zeolite molecular sieves, these being prepared in accordance with the disclosure in British Patent specification No. 1,382,896. Tap water at a temperature of 60°F and ice cubes were added to the glass and after 30 seconds, 90% of the flavor particles had completely dissolved, as measured by a refractometer, with the only agitation provided by the effervescence of the molecular sieves, and after 1 minute, a fully carbonated, cold, flavorful orange beverage was produced.

A flavorful, sweetened orange drink can also be obtained after one minute when 25 grams of the dry flavor particles are placed in an 8-ounce glass and tap water and ice cubes added to the mix. The agitation provided by the addition of tap water is sufficient to cause dissolution of the flavor particles and a uniform concentration of flavor in the final beverage.

EXAMPLE II

Fifty-seven parts of monosodium orthophosphate was dissolved in 440 parts distilled water. This mixture was added to 312 parts of a commercially available 2-ounce cola flavor base, to which 15 parts of 85% phosphoric acid had been added. The resulting mixture was then added to 80000 parts of granulated sugar (baker's sucrose) to form a slurry having a total solids content of about 93% and a flavor base: sugar ratio of about 0.039:1. This slurry was spread about 1/4" thick in freeze dryer trays and cooled to −10°F, at which temperature the slurry was frozen. The frozen slurry was then dried in a Stoke's freeze dryer in three temperature increments of −50°F, +50°F and 75°F for 1/2 hour, 4 hours and 2-1/2 hours, respectively, to form a dry, stable foam. This foam was gound in a two-roll mill and screened to obtain particles of the desired size range.

Twenty-five grams of the resulting dry beverage flavor mix and an operative amount of aluminosilicate molecular sieves, loaded with CO_2 (in accordance with the disclosure in British Patent Specification No. 1,382,896), were placed in an 8-ounce glass. Tap water

at a temperature of 60°F and ice cubes were added to make an 8-ounce beverage serving. The molecular sieves immediately began to effervesce and 90% of the flavor and sugar particles were fully dissolved within 30 seconds, as measured by a refractometer, and all flavor and sugar particles were completely dissolved within one minute. The only agitation was provided by the effervescence of the aqueous solution being carbonated by the molecular sieves. After the 1 minute mixing time, a flavorful, fully carbonated cola beverage resulted which appeared to be equal in carbonation and intensity of acid taste to cola beverages obtained from commercially available pre-carbonated bottled colas.

WHAT WE CLAIM IS:—

1. A process for preparing a rapidly dissolvable dry beverage flavour mix comprising:—

(a) forming a mixture of a beverage flavour base and sugar such that the mixture has a weight ratio of flavour base: sugar of from 0.03:1 to 0.15:1 and a total solids content of from 75% to 95% by weight;

(b) drying the mixture to form a stable foam; and

(c) grinding the stable foam to form discrete dry beverage flavour particles.

2. The process of claim 1 wherein the total solids content of the mixture is from 85% to 94% by weight.

3. The process of claim 1 or claim 2 wherein the mixture is dried to a final moisture content of less than 3% by weight.

4. The process of claim 3 wherein the mixture is dried to a final moisture content of less than 1% by weight.

5. A process for preparing a rapidly dissolvable dry beverage flavour mix substantially as herein described with reference to either of the Examples.

6. A dry beverage flavour mix whenever prepared by a process according to any of the preceding claims.

For the Applicants,
CARPMAELS & RANSFORD,
Chartered Patent Agents,
43 Bloomsbury Square,
London WCLA 2RA.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1977
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.